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CHARACTERIZATION OF IRON ORE PELLETS BASED ON MAGNETITE CONCENTRATES FROM SHERMAN MINES, ONTARIO

D.H.H. Quon and K.E. Bell
Mineral Processing Laboratory

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CHARACTERIZATION OF IRON ORE PELLETS BASED ON MAGNETITE
CONCENTRATES FROM SHERMAN MINES, ONTARIO

By

D.H.H. Quon* and K.E. Bell**

ABSTRACT

This paper discusses the microstructure of magnetite pellets supplied by Mineral Dressing Section, CANMET, which were sintered in air at 1300°C for 5 minutes. The bonding mechanism of the sintered pellets containing no additive with those containing calcium and ammonium chlorides are compared. Regardless of the presence or absence of the additives, the bonding characteristics of these pellets are similar.

Three of the most important parameters that affect the induration of the magnetite pellets, viz., temperature, time and additive content are assessed in terms of internal pellet structure and commonly-used pellet quality criteria.

*Research Scientist and **Head, Ceramic Section, Mineral Processing Laboratory, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa.

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INTRODUCTION

Iron ore production in Canada was approximately 45 million tonnes in 1975 of which 36 million was exported. It is apparent that iron ore makes a very important contribution to the maintenance of a favourable foreign trade balance. The most important aspect of the industry's development, apart from the growth in production, is the use of beneficiated iron ore, particularly pellets. Another aspect of the development is the removal of the undesirable constituents such as alkalies from the iron ore concentrates so that they are suitable for special metallurgical application.

As part of the current research program in CANMET, work has been directed towards the study of alkali removal from the iron ore concentrates, pelletizing of iron ore, and induration of iron ore pellets. In the iron ore process, variables such as raw materials, binder, water content in the green pellets, temperature and time of induration of the pellets, can affect the final strength of the iron pellets. It is the aim of this study to examine the effects of these variables on the properties of the finished ore pellets. The ultimate aim of this work is to establish optimum parameters for the production of finished ore pellets having acceptable properties to withstand compression and abrasion during shipping and to retain both physical strength and shape on entering the blast furnace for smelting.

The effect of temperature and induration time on compressive strength of the hematite iron ore pellets from Carol Lake, Newfoundland and Knob Lake, Quebec has been investigated earlier and the bonding mechanism of the iron ore pellets has been described in detail (1).

In the present investigation, the bonding characteristics of the pellets fabricated from the magnetite concentrates are examined. It has been reported by other investigators that the induration characteristics of the pellets of magnetite are appreciably different from the pellets of hematite (3,4).

EXPERIMENTAL

In this study, experimental work was conducted mainly in characterizing the sintered iron ore pellets based on the magnetite concentrates from Sherman Mines supplied by J. Wilson of the Mineral Processing Laboratory of

CANMET. According to Wilson the green pellets used for the sintering work contained less than 1 mass % of bentonite binder and/or less than 1 mass % of either calcium or ammonium chloride. The binder and additive concentrations in the green pellets are given in Table 1. The main aim of his study was to examine the effect of additive on the alkali removal from the iron ore concentrates. Prior to the induration of the pellets, they were dried in an oven at about 120°C followed by calcination at 850°C for 5 minutes. The final induration of the pellets was carried out in an electrical resistance furnace at a temperature of 1300°C for 5 minutes.

The as-received pellets were characterized by means of optical microscopy. Selected samples were also examined by scanning electron microscopy (SEM). Differential thermal (DTA) and thermogravimetric (TGA) analyses were conducted on green pellets in order to determine the physical and chemical changes taking place in the pellets with increased temperatures. The effect of temperature on the shrinkage of the green pellet was examined by means of a high temperature microscope. The various phases formed in the sintered pellets were determined by the X-ray diffraction technique (XRD).

RESULTS AND DISCUSSION

DTA and TGA results for the green magnetite pellets containing bentonite binder are given in Fig. 1. The TGA curve shows a weight loss from room temperature to about 200°C amounting to about 0.8 mass % due to the loss of physisorbed and chemisorbed water. Further increase in temperature caused a gain of about 1.6 mass % from 380 to 900°C. This was followed by a weight loss at a still higher temperature. The low temperature weight gain is due to the oxidation of magnetite to hematite. The high temperature weight loss is due to the decomposition of the carbonates present in the iron ore concentrate.

DTA curves for the green pellets as-received and dried in an oven at 120°C are given in Fig. 1. Essentially, these two curves are almost identical. The endothermic peaks at about 180 and 200°C are associated with the elimination of structural water in the bentonite binder. The endothermic peak occurring at 570°C is due to the transformation of alpha to beta quartz. The presence of some carbonates in the green pellet is represented by the very weak endothermic peak at about 780°C. The exothermic peaks recorded at 400

to 700°C are presumably related to either oxidation of the magnetite to hematite and/or due to the crystal growth of hematite.

X-RAY DIFFRACTION

The major phase in the sinters is hematite with or without magnetite, as indicated by the XRD. Other phases detected are quartz and cristobalite.

HEATING MICROSCOPY

The effect of temperature on the shrinkage of iron pellets was determined by heating microscope. A 2 x 2 x 2 mm specimen was prepared from a green pellet dried at 100°C. This sample was loaded into the heating microscope furnace and heated to 1400°C using a heating rate of approximately 6°C/min. The change in specimen dimension due to increase in temperature was photographically recorded at temperature intervals of about 100°C. In this manner, a series of photographs was obtained showing progressive shrinkage and deformation of the sample; the photographic negatives were first enlarged and the projected image was traced on paper. The image obtained from tracing was then cut out from the paper and subsequently weighed. Assuming the paper was of uniform thickness, the progressive weight change of each trace is directly proportional to the progressive area change. The results of the progressive shrinkage and deformation of the magnetite pellet with temperature are plotted in Fig. 2.

On heating the green magnetite pellet, the sample shows four sharp discontinuous shrinkage peaks at 300, 425, 750 and 1400°C. Each stage of shrinkage is immediately followed by a slight expansion. The first two shrinkages are attributed to dehydration of the adsorbed water and dehydroxylation of the bentonite. The shrinkage that occurs at 750°C is due to the decomposition of the carbonates, which are present as a gangue mineral in the iron ore concentrates. The shrinkages followed by a series of expansions have been attributed to the mechanical bloating caused by internal pressures generated by the gases released. The most distinguishable expansion of the pellet was recorded at about 1100°C. This expansion is associated with the rapid grain growth of the hematite, which commences at about 1000°C. Followed by the expansion, the sample began to shrink and reached the maximum at about 1400°C. This shrinkage is attributed to the densification of the hematite grains, which increases with increasing temperature.

A limited amount of experimental work was conducted to determine the rate of shrinkage of the iron ore pellet under conditions simulating the sintering process. In this case a small sample of a green pellet was pre-sintered at 850°C for 5 minutes. It was then rapidly introduced into the sintering zone at 1275, 1300 and 1350°C for a time varying from 5 to 20 minutes. The shrinkage of these samples was again recorded photographically, and the technique for obtaining the shrinkage was carried out in the same manner as described earlier. The shrinkage versus time at different temperature for the green pellet samples are shown in Fig. 3. As can be seen from the figure, at a sintering temperature of 1275°C the ideal sintering time is 20 minutes. At 1300°C, a sintering time of 5 minutes is sufficient to reach reasonable density. With further increase in sintering time there appears to be a further shrinkage of the sample, but this results in an increase in the amount of liquid phase developed, which further reduced the hematite to magnetite.

MICROSTRUCTURAL ANALYSIS

Polished sections were prepared and examined using an optical microscope. Selected samples were also examined by means of scanning electron microscopy (SEM); semi-quantitative analysis for Ca, K, Al and Si was obtained by X-ray mapping using the energy dispersive technique (EDXR).

Typical microstructures of the sintered pellets are shown in Fig. 4 to 9. It is apparent from the micrographs that all pellets have a common characteristic microstructure and the hematite grains are bonded together by neck growth with numerous pores being formed between the grains. The effect of additive in microstructure modification is not readily noticeable. There is a tendency for the hematite to form a fine grained matrix. Nevertheless, there is some scattered, poorly bridged coarse-grained hematite also present. In some samples, magnetite is commonly observed at the core of the pellet and this gives the appearance of a shell structure. There is a wide range of grain size distribution in the sinters. In some cases, coarse-grained hematite of 20 to 50 μm size occurs in a fine-grained hematite matrix of less than 5 μm in size. The bridging of the fine grained hematite is less well developed than the coarse-grained hematite. Under crossed polars, the coarse-grained hematite appears as lath-shaped crystals with minor pores present at the grain boundaries. In some sinters a modification of grain was evident by the slightly rounding off of the grains. This indicates that some liquid

phase was present and the rounding off of the grain occurred through the dissolution of the hematite in the liquid phase. However, extensive development of the liquid phase in the sinters is not common. There is a tendency for sinters containing ammonium chloride additive to develop a slightly finer grained and higher porosity structure than sinters containing calcium chloride additive. It is reasonable to speculate that the high porosity and finer grains in the sinters containing small amounts of ammonium chloride are related to the decomposition of the ammonium chloride during the presintering stage. The volatiles tend to escape at the decomposition temperature of ammonium chloride and force the grains apart and reduce the grain to grain contact, which makes it difficult to indurate at high temperature. In sinters doped with calcium chloride the hematite tends to develop into coarser grains with lower porosity. This is due to the ability of the calcium to react with the other phases in the magnetite concentrates to form a liquid phase, which fills the pores formed in the sinter.

The presence of magnetite in the sinters, as mentioned earlier, is a common phenomenon. This indicates that some of the magnetite has not been sufficiently oxidized prior to the final sintering temperature. The shelling effect is extremely undesirable as this would lead to degradation of pellet strength. In order to decrease the shelling in the sinters, a close control of the sintering steps must be exercised. It is important to give sufficient time for the oxygen to diffuse into the pellet at temperatures below 1000°C prior to the final sintering at 1300°C in order to complete the oxidation of the magnetite to hematite. Figure 10 shows a typical example of a magnetite core formed in the sinters. As can be seen from the figure the magnetite is rimmed by a thin layer of hematite and also intergrown with hematite. Based on the above observation it can be assumed that these pellets were introduced into the sintering zone too fast. The outer shell of the pellet only was oxidized and the inner shell stayed intact. Other factors that may prevent the complete oxidation of the sinters are the consolidation of the hematite grains developed first at the outer shell and the reduction of both pores and grain boundaries in the material, which hinder the movement of the oxygen into the interior of the pellet.

SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy (SEM) was used to examine samples sintered at a temperature of 1300°C for 5 minutes.

A SEM micrograph of a sample doped with 1 mass % calcium chloride is shown in Fig. 11. The size of hematite grains varies from about 1 to 40 μm . The large hematite "grains are composed of numerous smaller grains and are bridged together by a" continuous grain growth with pores being trapped at the grain boundaries and showing very little evidence of the presence of a slag phase. In addition to the hematite grains, other phases detected in the sample are quartz with minor amounts of other phases. Qualitative analysis of the minor phases by EDXR shows that these are solid solutions containing K, Al, Fe, Si and Mg. These phases form intergrowth and exsolution lamellae and are commonly associated with the quartz grains. Elemental distribution of Ca, Si, Al and K for this sample are given in Fig. 12 to 15. The results show that the amount of calcium present in the sample is very low even with a doping concentration of 1 mass %. The silica distribution can be readily recognized as it tends to associate with the quartz grains. Aluminum is distributed mainly in the minor phases containing exsolution or intergrowth of K, Al, Fe, Si and Mg. Potassium occurs either in the minor intergrowth or in the exsolved phases or at the grain boundaries of the hematite.

The microstructure of the sample doped with 0.5 mass % ammonium chloride is shown in Fig. 16. Elemental distributions of Al, Si and K in this sample are shown in Fig. 17 to 19 respectively.

Figure 20 shows the microstructure of the sinter containing no dopants; EDXR maps for Al, Si and K are given in Fig. 21 to 23 respectively.

Based on the results obtained from the SEM examination, it may be concluded that the microstructures of the sinters with or without doping are essentially similar. The presence of quartz in these sinters indicates that the original quartz grains in the iron ore concentrates were not reacted at high temperature. The formation of both intergrowth and exsolution textures containing K, Al, Si, Fe and Mg is due to the reaction of the accessory minerals in the iron ore concentrates.

GRAIN SIZE MEASUREMENTS

In the previous section, the grain size of the sintered pellets was described as varying from a few micrometers to about 50 micrometers. In

order to obtain the grain and pore size distribution in the samples, a Zeiss Videoplan* computerized image analysis was used. Photomicrographs of the sinters were obtained from the optical microscope at a magnification of 1250x. The dimensions of the grains and pores in the photomicrographs were analyzed by the image analyser and the different size fractions of the grains and pores were calculated and classified by the computer. The results obtained are given in Table 2 and they confirm the earlier observation that the sinter containing ammonium chloride additive developed finer grains as compared with the samples containing bentonite and/or bentonite with calcium chloride. It is rather difficult to draw any conclusion as to how sintering temperature and time affect the grain growth and development porosity as these samples were sintered at constant temperature and time. Also, other process variables added further complication to the interpretation of the above results.

An examination of the effect of sintering temperature and time on the bonding mechanism of the pellets was conducted. In these experiments, magnetite pellets containing 1 mass % of bentonite were prepared. The green pellets were sintered at temperatures ranging from 1150 to 1300°C for a period ranging from 5 to 20 minutes. Polished sections of these samples were prepared, and photomicrographs taken. Again the Videoplan image analysis was used to determine the grain and pore size distribution under different sintering conditions. The results are given in Table 3. As can be seen from the table, the hematite formed intergrowths at a temperature as low as 1150°C and these intergrown grains reached size levels up to 172 micrometers. An increase in the sintering temperature and time tends to decrease the grain size. This apparent decrease in the grain size is due to the densification of the intergrown grains at higher sintering temperatures.

BONDING MECHANISM OF THE MAGNETITE PELLETS

Based on the DTA, TGA, heating microscope and microstructural examination of the pellets sintered under various conditions, major changes occur to the pellet constitution as the temperature is raised to approximately 1000°C in the preheating stage of the firing cycle. Typical reactions occur-

*Manufactured by Carl Zeiss Ltd., West Germany

ring during the preheating stage are: dehydration of adsorbed water, dehydration of bentonite and decomposition of dopants such as ammonium and calcium chlorides. Also in the preheating stage, at a temperature well below 1000°C, the magnetite gets oxidized or decomposes to form hematite prior to the final sintering temperature. The degree of decomposition or oxidation from magnetite to hematite is controlled by the rate of heating as well as the temperature of heating. During this oxidation, the factors that influence the microstructure of the sintered products include the temperature of sintering, the air flow velocity, the partial pressures of the heating gases and the grain shape and size of the concentrates. The porosity of dry pellets plays a certain role as it provides for oxygen to penetrate into the core of the pellet. Insufficient oxygen to oxidize the magnetite would cause the formation of a core structure having a center core of magnetite surrounded by an outer shell of hematite. Shelling structure can also be brought about by rapid heating of the magnetite pellet from a low temperature to a higher sintering temperature. Under this condition, the grain growth tends to occur first at the surface of the pellet and simultaneously the heat being generated inside the pellet due to oxidation increases the core temperature of the pellet. The rise in temperature coupled with grain growth results in the reduction of porosity and this prevents oxygen penetration to the interior of the pellet, thus retaining the magnetite unoxidized. The control of firing velocity to match oxidation velocity has a positive effect during firing of the magnetite pellets. Unless these two parameters are optimized, it is not surprising to find shelling structures in such sinter products. The shelling structure results in considerable pellet quality impairment and is undesirable.

A considerable difference in sintering mechanism for the iron ore concentrates of hematite and magnetite has been observed. In the sintering studies dealing with the hematite and specular hematite pellets from Knob Lake and Carol Lake respectively, the following bonding mechanisms have been proposed:

1. Low temperature sintering (below 1300°C).
 - (a) Induration of fine hematite particles.
 - (b) Continuous and discontinuous grain growth.
 - (c) Very minor or no liquid phase sintering.
2. High temperature sintering (above 1300°C).
 - (a) Continuous and discontinuous grain growth.
 - (b) Bridging of grains (neck growth).
 - (c) Liquid phase sintering (slag bonding).

During the sintering of the magnetite pellets as observed from the microstructural analyses, the sequence of induration of the iron ore concentrates is the following:

1. Low temperature induration (below 1100°C).
 - (a) Oxidation of magnetite to fine-grained hematite.
 - (b) Continuous and discontinuous grain growth of the fine-grained hematite.
2. High temperature induration (above 1200°C).
 - (a) At lower temperature both continuous and discontinuous grain growth and neck growth of the larger continuous and discontinuous grains.
 - (b) Development of liquid phase at a temperature in the vicinity of 1300°C as evidenced by the slight modification by rounding of the coarse-grained hematite.

The high strength of the pellets from the magnetite concentrates obtained by sintering at a temperature substantially lower than the hematite concentrates may be attributed to the oxidation reaction of the former material during the pre-sintering step, which increases the surface area of the particles. The slag bonding of the pellets is absent, with the exception of a few samples where a liquid phase was observed. Its presence is due to the melting and reaction of the accessory minerals such as feldspar, mica, amphibole and pyroxene with the iron ore concentrates. These accessory minerals are reported to be present in the magnetite concentrate (2).

CONCLUSIONS

Three important parameters, viz., temperature, time and additive content, influence the internal pellet structure and commonly-used pellet quality criteria of iron ore pellets. They are characterized by non-equilibrium mineral assemblages as reflected by the presence of a magnetite core in some sinters due to inhibiting the oxidation of magnetite at high temperatures.

The bonding characteristics of the magnetite pellets are different from those of the hematite pellets. In the sintering of the magnetite from the Sherman mine, the magnetite was first oxidized at a temperature below 1000°C to hematite. The strength development of the magnetite pellets is ap-

preciably higher than the hematite pellets under similar induration conditions due to the conversion of magnetite to hematite at this stage. The higher strength of the magnetite pellets is due to the higher surface areas resulting from the oxidation, which renders the material more reactive.

Based on the microstructural examination, the bonding of the magnetite pellets is basically by grain growth with very little slag bonding. The effect of additives such as ammonium or calcium chlorides on the bonding characteristics is minimal.

The presence of a magnetite core in the partially oxidized pellets indicates that the diffusion of oxygen into the core of the pellet was hindered by the decreased porosity at the external shell of the pellet. The coring may affect the pellet strength due to the hematite and magnetite heterogeneous bond. In order to reduce the coring effect, the magnetite pellets must be allowed to oxidize at a temperature below 1000°C.

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Table 1 - Doping concentration and moisture content of green pellets

Sample No.	Dopant, Mass%			Moisture, %
	bentonite	NH ₄ Cl	CaCl ₂	
SH2501-1	0.7			8.1
SH2501-2	0.7			8.3
SH2701-1	0.7			8.6
SH2801-1	0.7		1.0	7.99
SH0202-1		1.0		-
SH0202-2		0.5		-
SH0102-1	0.7		0.5	8.6
SH0302-1	0.7			-
SH0502-1EX	1.0			9.7
SH0802-1	1.0		1.0	8.0
SH0902-1	1.0		0.5	8.45
SH1002-1	1.0		1.0	7.65
SH1102-1	1.0	0.5		8.15
SH1202-1	1.0	1.0		-
SH2602-1	+ 1.0	0.5		8.47
SH0103-1	+ 1.0	0.5		-
SH0203-1	+ 1.0		1.0	9.3
SH0203-2	+ 1.0		0.5	9.3
SH0403-1	+ 0.5			-
SH0403-2	+ 0.5		1.0	-
SH0503-1	+ 0.5		0.5	8.57
SH0803-2	+ 0.5	1.0		8.07
SH0903-1	1.0			9.10
SH1103-1	1.0	1.0		9.10
SH1203-1	0.5		1.0	7.50
SH1603-1	0.75			9.50
SH1703-1	0.75		1.0	8.17
SH1803-1	0.75		1.0	8.41
SH2503-2EX *	1.0		0.5 + 0.5 FeCl	7.50
SH3003-1 *	1.0		0.5 + 0.5 FeCl	-
SH3103-1 *	0.75	1.08		9.10
SH3103-2 *	0.75	0.5		

+ Sintered in small electrically heated furnace.

* Sintered in a gas-fired furnace.

Table 2 - Grain and pore size in the as-received sintered pellets containing various dopings

Sample No.	Bentonite, mass %	Doping, mass%		Grain size, μm			Pore size, μm		
		NH_4Cl	CaCl_2	Min.	Max.	Mean	Min.	Max.	Mean
SH0802-1	1.0	0	1.0	3.2	74	22	5	41	18
SH1002-2	1	1	0	1	10	4	1	11	0.3
SH2501-2	0.7	0	0	1.4	32	6.4	1	12	3
SH0102-1	0.7	0	0.5	5.0	119	29.0	5	38	15
SH2501-1	0.7	0	0	4.6	81	27.0	2.4	40	14
SH0202-1	0.7	0.7	0	0.7	27	4.5	0.6	17	3.6

* All pellets were sintered at 1300°C for 5 minutes.

Table 3 - Grain and pore size development vs. sintering temperature and time*

Sample No.	Sintering condition		Grain size, μm			Pore size, μm		
	temp, °C	time, min.	min.	max.	mean	min.	max.	mean
1	1150	5	8.1	172	43	4	57	13
2	1175	5	7.8	97	34	1.4	55	8
3	1200	5	5.9	67	24	6.6	46	20
4	1300	5	1.5	22	7	0.8	34	5
5	1250	10	1.7	18	5	0.6	7	3
6	1250	20	0.9	33	5	0.6	10	3
7	1275	5	1	18	4	1.3	18	3
8	1275	10	1.5	13	5	1	10	3
9	1275	20	0.7	16	5.3	1	12	3

* Pellets containing 1 mass % bentonite binder.

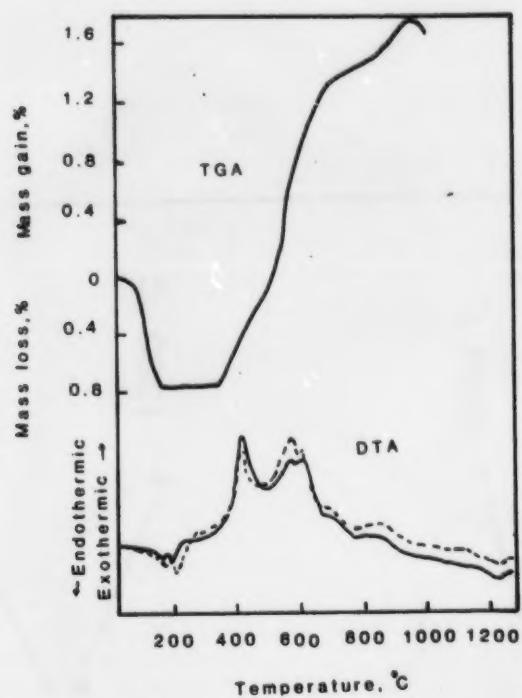


Fig. 1 - DTA and TGA of the green pellet

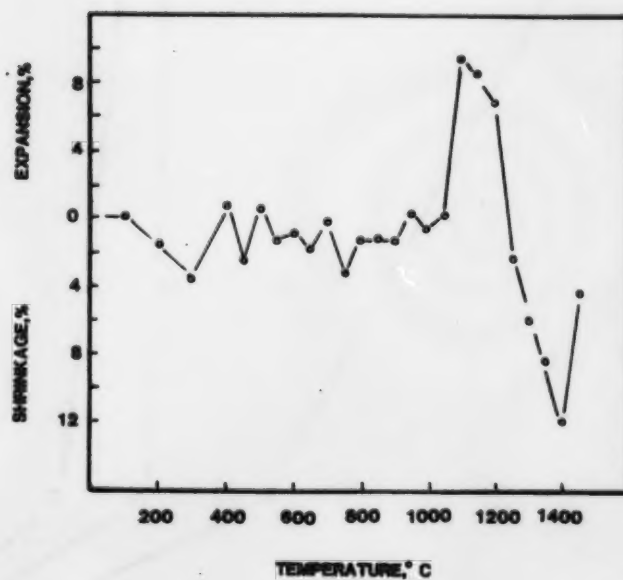


Fig. 2 - Shrinkage and expansion of green pellet as observed by heating microscopy.

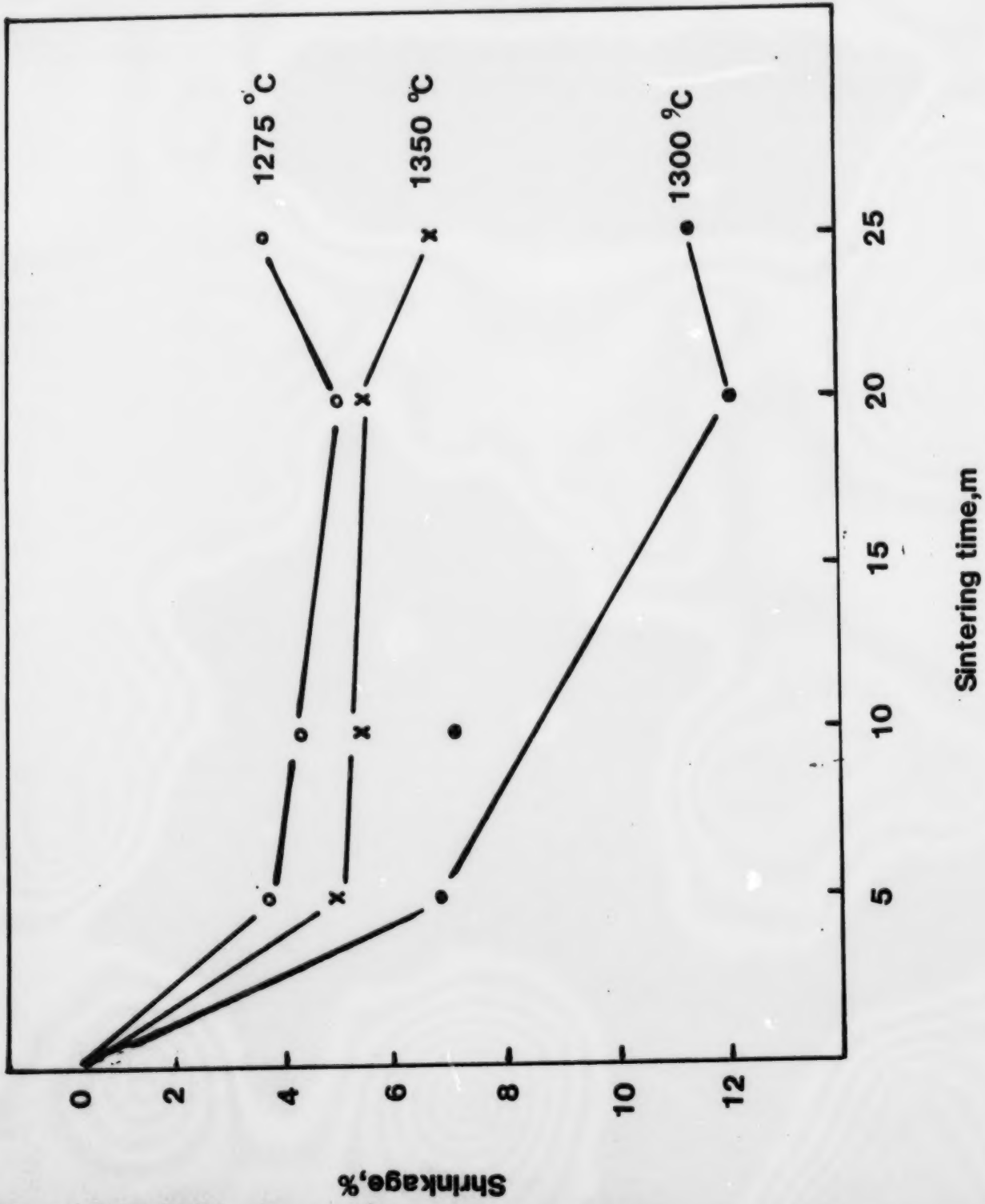


Fig. 3 - Shrinkage and expansion of green pellet versus temperature and time.

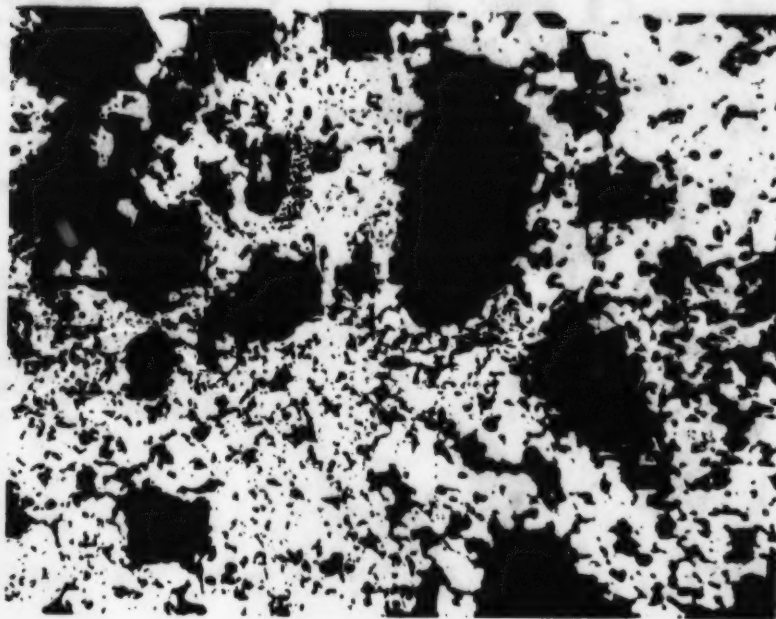


Fig. 4 - Microstructure of pellet containing 0.7 mass % bentonite sintered at 1300°C for 5 min.

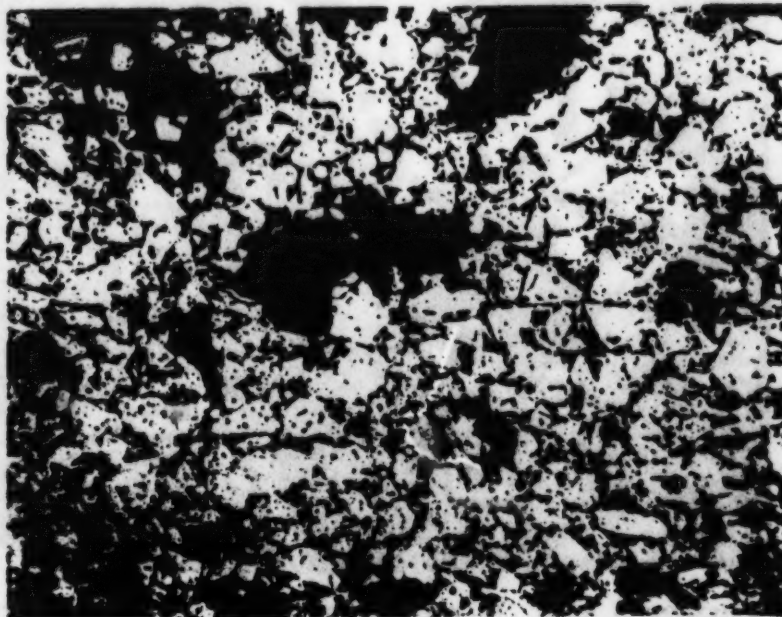


Fig. 5 - Microstructure of pellet doped with 1 mass % CaCl_2 and 1 mass % bentonite sintered at 1300°C for 5 min.

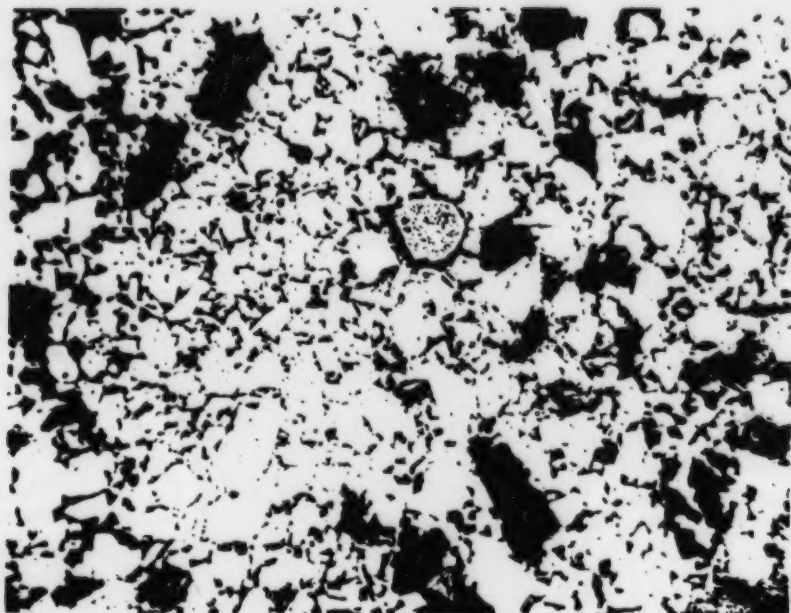


Fig. 6 - Microstructure of pellet doped with 1 mass % NH_4Cl and 0.7 mass % bentonite sintered at 1300°C for 5 min.

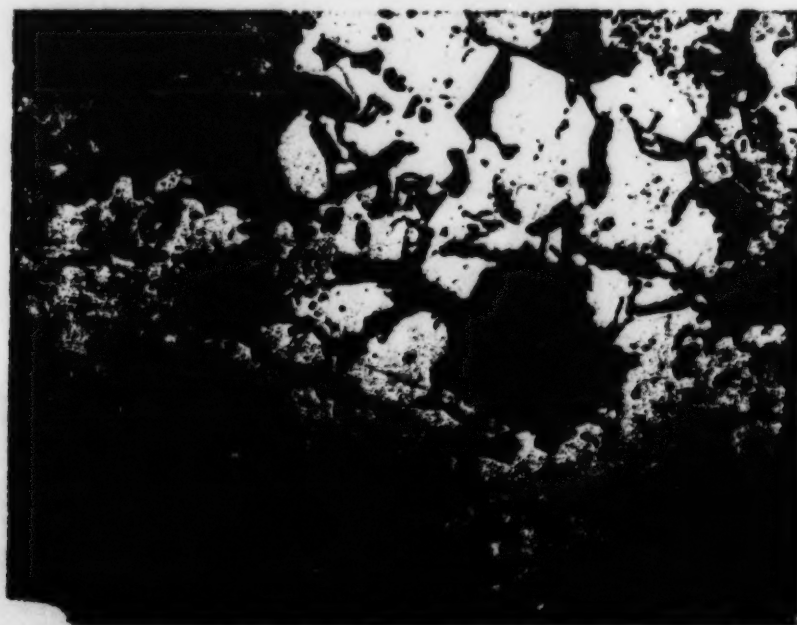


Fig. 7 - Microstructure of pellet containing 0.7 mass % bentonite sintered at 1300°C for 5 min. (higher magnification of Fig. 4).

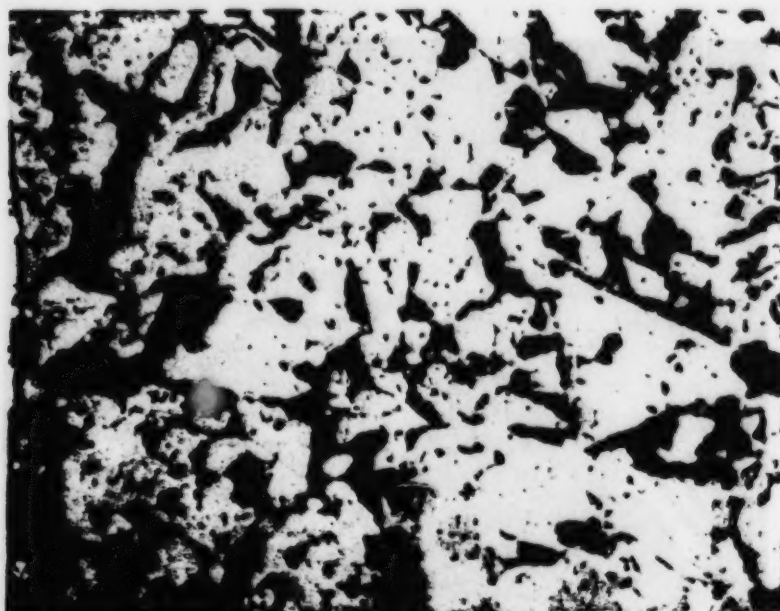


Fig. 8 - Microstructure of pellet doped with 1 mass % CaCl_2 and 1 mass % bentonite sintered at 1300°C for 5 min. (higher magnification of Fig. 5).

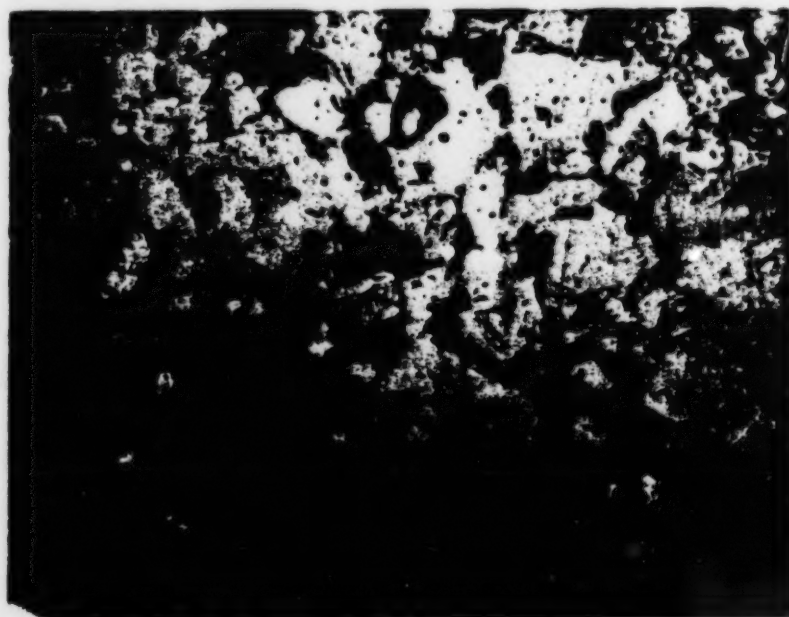


Fig. 9 - Microstructure of pellet doped with 1 mass % NH_4Cl and 0.7 mass % bentonite sintered at 1300°C for 5 min. (high magnification of Fig. 6).

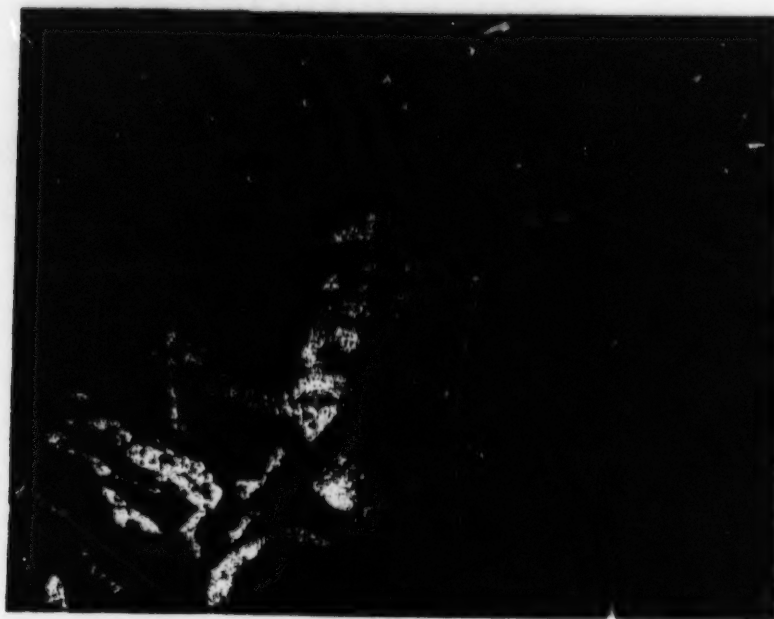


Fig. 10 - Magnetite grains observed in the core of the sintered pellet.

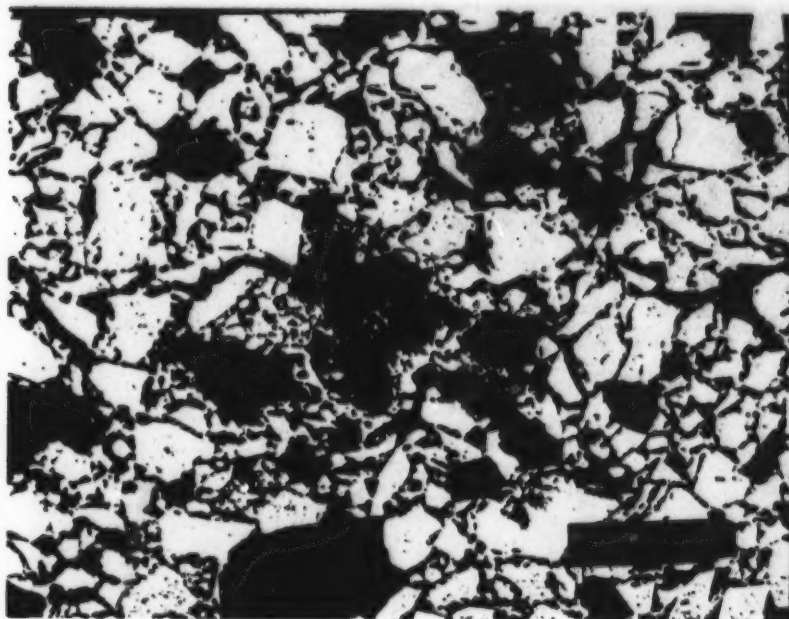


Fig. 11 - SEM micrograph of pellet doped with 1 mass % CaCl_2 and 0.5 mass % bentonite sintered at 1300°C for 5 min.



Fig. 12 - EDXR map showing K distribution for SEM micrograph Fig. 11.



Fig. 13 - EDXR map showing Al distribution for SEM micrograph Fig. 11.



Fig. 14 - EDXR map showing Si distribution for SEM micrograph Fig. 11.

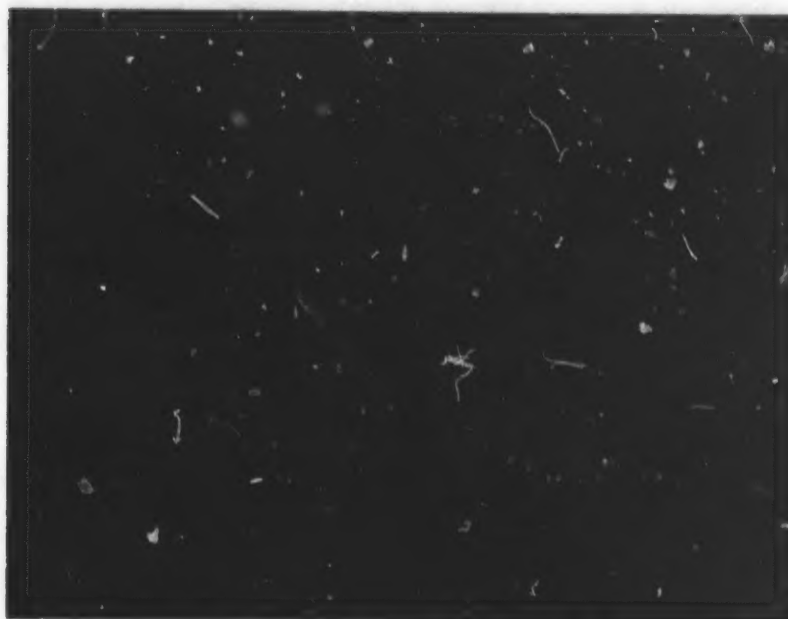


Fig. 15 - EDXR map showing Ca distribution for SEM micrograph Fig. 11.

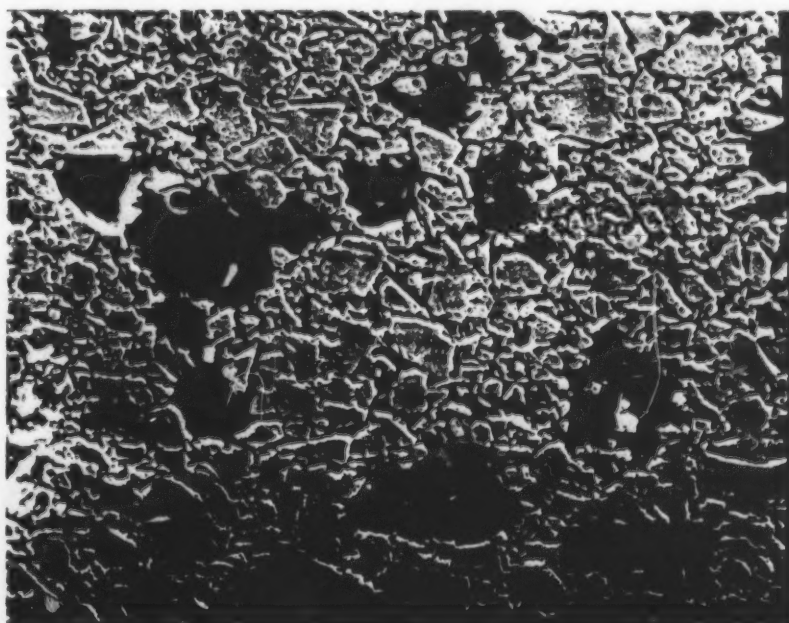


Fig. 16 - SEM micrograph of pellet doped with 0.5 mass % NH_4Cl and 0.7 mass % bentonite sintered at 1300 C for 5 min.



Fig. 17 - EDXR map showing K distribution for micrograph Fig. 16.



Fig. 18 - EDXR map showing Al distribution for micrograph Fig. 16.



Fig. 19 - EDXR map showing Si distribution for micrograph Fig. 16.

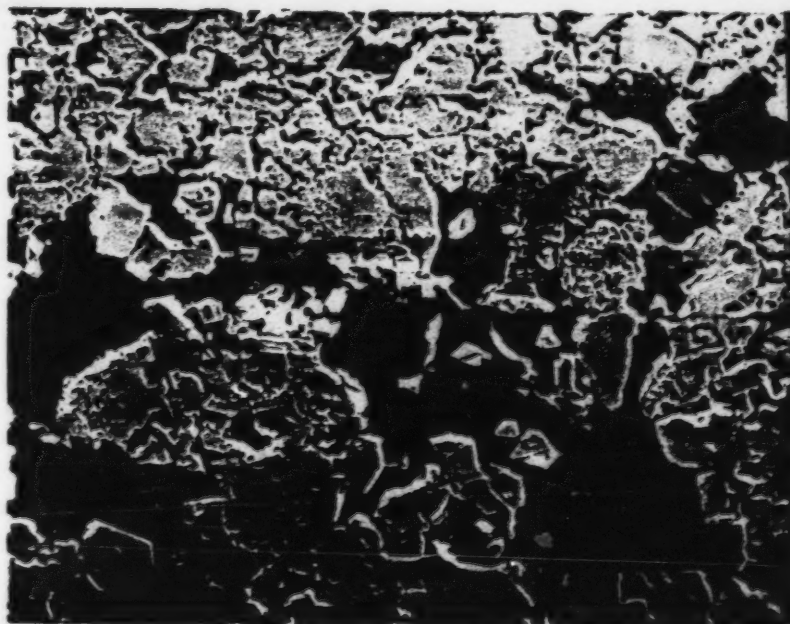


Fig. 20 - SEM micrograph of pellet containing 0.8 mass % bentonite sintered at 1300°C for 5 min.



Fig. 21 - EDXR map showing K distribution for micrograph Fig. 20.



Fig. 22 - EDXR map showing Al distribution for micrograph Fig. 20.



Fig. 23 - EDXR map showing Si distribution for micrograph Fig. 20.